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DETECTION OF LIME USED AS A NEUTRALIZER IN DAIRY PRODUCTS.

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INTRODUCTION.

The use of lime by central-station creameries before pasteurization for the purpose of reducing the acidity of cream before churning has assumed considerable proportions. The cream is bought directly from farmers at the various cream stations and then shipped in an unneutralized state to the centralizing creamery to be manufactured into butter. On arrival it is graded into two classes, according to its taste and smell rather than its acidity. As the cream often has been shipped several hundred miles to the central plant, it frequently arrives in a very old and sour state, sometimes with an acidity of over 1 per cent lactic acid. Cream with a "bad" taste or foreign odor is churned into second-grade butter. If the cream is too sour, lime is added in order to reduce the acidity to 0.3 or 0.4 per cent lactic acid. The creameries claim to be able to make a good grade of butter even from a very sour cream by the use of lime, and the butter thus made is said to grade a number of points higher than that made from the same cream not neutralized. Hence it has become a common practice, especially in summer when it is difficult to

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NOTE.—This bulletin will be useful to officials of State dairy and food departments.

prevent excessive souring, to "lime" or "neutralize" cream of high acid content, before pasteurization, in order to reduce its acidity and improve the salable quality of the butter made from it. Some creameries use sodium carbonate for this purpose instead of lime.

The work reported in this bulletin was undertaken for the purpose of developing and perfecting a method for detecting with certainty the addition of lime to cream before its manufacture into butter. It is believed that no such method has heretofore been published. The detection of alkali salts when used to neutralize dairy products has not been considered in this publication but has been left for future work. Nor has any consideration been given to the question of possible detriment to health, or to the legal, economic, and ethical phases of the process discussed in this bulletin.

Milk and products derived from milk contain calcium phosphate as one of the principal constituents of the ash. Leach[1] states that the percentage of calcium oxid in the ash of a typical milk is 20. Blyth[2] gives figures ranging between 17.31 and 27.55 with a mean of 22. Tibbles[3] quotes an analysis by Schrodt wherein the calcium oxid is 21.45 per cent of the ash. König[4] gives a figure of 22.42 per cent. Köstler[5] reports values for the percentages of calcium oxid in the ash of milk, cream, buttermilk, and butter. His maximum and minimum figures are given in Table 1.

TABLE 1.—*Percentages of calcium oxid in the ash of dairy products, as calculated by Köstler.*

Substance.	Number of analyses.	Calcium oxid in ash.	
		Maximum.	Minimum.
		Per cent.	*Per cent.*
Milk	10	24.63	22.24
Skimmed milk	1	24.48	....
Cream	1	23.09	....
Buttermilk	19	24.54	21.22
Butter	11	22.83	20.07

Shaffer and von Fellenberg[6] found values between 18.3 and 20.2 per cent for butter.

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[1] Leach, Albert E., Food Inspection and Analysis, 3d ed., p. 128, New York, 1913.

[2] Blyth, A. W. and M. W., Foods: Their Composition and Analysis, p. 201, London, 1909.

[3] Tibbles, William, Foods, Their Origin, Composition, and Manufacture, p. 245, London, 1912.

[4] König, J., Chemie der Menschlichen Nahrungs- und Genussmittel, 4th ed., v. 2, p. 603, Berlin, 1904.

[5] Köstler, G. Zur Charakterisierung unserer schweizerischen Butterarten. *In* Landw. Jahrb. der Schweiz, 25 (1911), 249–276.

[6] Shaffer, F., and von Fellenberg, Th. Zur Unterscheidung der Butterarten. *In* Mitt. Lebensm. Hyg., 2 (1911), 220.

While it is probable that the percentage of calcium oxid in the ash of pure cream and butter would correspond closely to the figures quoted above, it is important to prove this point before proceeding with the interpretation of analyses of commercial milk products to determine whether lime may have been added.

METHODS OF ANALYSIS.

Samples of milk, buttermilk, or cream require no particular preparation for analysis. All samples of butter analyzed in this investigation were prepared in the following way: About 1 pound of butter was melted at as low a temperature as possible in a well-stoppered, wide-mouthed bottle and then cooled. The melted fat, curd, water, and salt were violently shaken together before and during the "setting" of the butter. If the sample subsequently is kept cool, it will not leak buttermilk, thus causing variations in sampling.

In order to prove the addition of lime to dairy products a careful analysis of the ash is essential. To determine total ash, 10 grams of milk, cream, or butter are weighed out in a platinum dish, the water evaporated on a steam bath or air oven, the fat then burned off, and the dish heated in a muffle at a low heat until a white ash remains. The water must be completely evaporated before the fat is ignited, otherwise there will be loss by spattering. In the case of salted butter, after weighing the ash add dilute nitric acid and determine the sodium chlorid by precipitation with silver nitrate. All results reported in this bulletin were determined gravimetrically, with the exception of alkalinities. Unless the percentage of salt is more than 5 it is best determined without taking an aliquot. The difference between the total ash and the sodium chlorid in the ash is the salt-free ash. In duplicate determinations there may be a noticeable difference in the total ash, but the percentages of salt-free ash agree very closely. Examples of some of the results obtained are given in Table 2. No effort was made to select especially favorable results.

Table 2.—*Agreement in percentages of salt-free ash in dairy products.*

Sample No.	Total ash.	Salt.	Salt-free ash.	Sample No.	Total ash.	Salt.	Salt-free ash.
	Per cent.	*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
1	2.117	2.004	0.113	6	3.430	3.285	0.142
	2.066	1.956	.110		3.400	3.258	.142
2	3.887	3.735	.152	7	3.840	3.694	.146
	3.863	3.718	.145		3.840	3.700	.140
3	4.860	4.660	.200	8	4.784	4.654	.130
	4.779	4.577	.202		4.758	4.629	.129
4	4.768	4.647	.121	9	4.780	4.620	.160
	4.640	4.518	.122		4.808	4.644	.164
5	4.847	4.675	.172	10	4.822	4.721	.101
	4.870	4.693	.177		4.753	4.655	.098

The salt-free ash in duplicate analyses should agree within 0.01 per cent, and further analysis should be made if the results do not agree within this limit.

Disagreements in the total ash are considered as due to volatilization of sodium chlorid at different temperatures of ignition and not to variation in sampling. If the differences were due to variation in sampling the ash of unsalted butter should show the same relative discrepancies. To show that this is not the case, ash percentages of three unsalted butters are given as follows:

TABLE 3.—*Agreement in ash percentages of unsalted butters.*

Sample No. 1.	Sample No. 2.	Sample No. 3.
Per cent.	*Per cent.*	*Per cent.*
0.139	0.202	0.088
.141	.204	.087
.137	.205	.086

The differences between triplicate determinations are well within the 0.01 per cent limit.

The amount of sample to be used in making the calcium oxid determinations will depend on the capacity of the dishes available or the quantity of ash present. In the experiments subsequently reported the ash of from 10 to 50 grams of sample was used, treated as follows: A known quantity of decinormal hydrochloric acid was added, warmed to dissolve the ash, and after cooling the solution was titrated back with decinormal sodium hydroxid to obtain the alkalinity, using Methyl Orange as the indicator. Acetic acid was then added in slight excess and the calcium precipitated from hot solution with ammonium oxalate. After standing overnight, preferably on a warm steam bath, the calcium oxalate was collected on a small filter, washed free from chlorid, and ignited to constant weight as calcium oxid. The volume of solution when filtered should not be over 100 cc. Under conditions of slight acidity and small volume the error in the calcium determination, due to the solubility of calcium oxalate in acetic acid solution, is negligible. When iron or aluminum is present the ferric or aluminum phosphate, being insoluble in acetic acid, must be filtered off before the precipitation with ammonium oxalate. Practically, this is necessary only in those cases where cream or butter has become contaminated with iron from the container. Leach[1] gives the percentage of iron oxid in the ash of milk as 0.13 and makes no mention of aluminum. The average ash of milk is 0.71 per cent, cream has less ash, depending on the amount of fat, and the ash of unsalted butter is usually less than 0.2 per cent. The amount of iron or aluminum found in the ash of uncontaminated products in the amounts taken for analysis is negligible. The following data, taken at random from a large number of determinations, show how closely duplicate determinations of calcium oxid will check:

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[1] Leach, Albert E., Food Inspection and Analysis, 3d ed., p. 128, New York, 1913.

TABLE 4.—*Agreement in percentages of calcium oxid in ash of dairy products.*

Percentage of CaO.	Percentage of CaO.	Percentage of CaO.	Percentage of CaO.	Percentage of CaO.
0.0360	0.0500	0.0575	0.0637	0.0772
.0372	.0500	.0600	.0642	.0765
.0410	.0280	.0356	.0425	.0567
.0405	.0285	.0340	.0410	.0562

These figures show that with careful work results can be obtained that check considerably within 0.003 per cent; 0.01 per cent for salt-free ash and 0.003 per cent for calcium oxid are considered the maximum differences allowable.

Samples of milk collected in Denver gave figures comparable to those found in the literature, as shown in Table 5.

TABLE 5.—*Analysis of Denver milks.*

Constituent.	Jersey milk.	Holstein milk.	Unknown milk.
Fat (per cent)	4.50	3.30	4.00
Ash (per cent)	0.80	0.75	0.73
Alkalinity of ash (cc N/10 acid per 100 grams)	78.00	66.50	68.50
Calcium oxid in milk (per cent)	0.194	0.165	0.17
Calcium oxid in ash (per cent)	24.20	22.00	23.30

ANALYSIS OF UNNEUTRALIZED CREAMS.

Analyses of a considerable number of creams received by local creameries were made at the Denver laboratory of the Bureau of Chemistry during the summer of 1913. The samples of cream, the ash data of which are given in Table 6, were secured before neutralization.

TABLE 6.—*Analysis of ash of Denver creams before liming.*

Ash.	Ash on fat-free basis.	Alkalinity of ash on fat-free basis.[1]	CaO in cream.	CaO in ash.	CaO on fat-free basis.
Per cent.	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
0.42	0.73	71.6	0.100	23.7	0.174
.47	.72	68.5	.106	22.5	.163
.51	.79	68.5	.105	20.5	.163
.44	.71	64.5	.100	22.7	.161
.49	.76	68.7	.111	22.6	.173
.35	.56	51.4	.077	22.0	.123
.45	.74	69.5	.100	22.0	.165
.46	.66	56.4	.102	22.1	.147
.45	.71	65.7	.100	22.2	.158
.31	.53	54.6	.077	24.8	.133
.40	.63	71.0	.095	23.7	.150
.39	.66	57.9	.088	22.5	.150
.31	.53	55.2	.077	25.0	.133
.39	.72	....	.091	23.3	.168
.46	.73	58.4	.111	24.1	.177
.36	.65	60.6	.085	23.6	.154
.40	.72	73.5	.097	24.3	.175
.44	.75	67.1	.100	22.7	.172
.45	.77	60.6	.104	23.1	.179
.48	.64	60.8	.117	24.3	.158
.62	.83	78.4	.144	23.2	.193
.50	.75	76.5	.120	24.0	.181
Average..0.43	0.69	66.3	0.100	23.1	0.161

[1] Cc N/10 acid per 100 grams (Methyl Orange, indicator).

From the figures given in Table 6 it would appear that the following are conservative maximum values for unneutralized cream:

Alkalinity of ash on fat-free basis (cc N/10 acid per 100 grams)	80.00
CaO in cream on fat-free basis (per cent)	0.20
CaO in ash (per cent)	25.00

ANALYSIS OF DAIRY PRODUCTS MADE FROM UNNEUTRALIZED CREAMS.

A number of analyses of butter churned from unneutralized cream were made at the Denver laboratory during the year 1913. The ash data are given in Table 7.

TABLE 7.—*Analyses of ash of butter made from unneutralized cream. 1913.*

Alkalinity of ash.[1]	CaO in butter.	Salt-free ash.	CaO in salt-free ash.
	Per cent.	*Per cent.*	*Per cent.*
12.0	0.026	0.13	20.0
13.3	.026	.16	16.3
10.6	.024	.12	20.0
14.5	.030	.15	20.0
8.5	.017	.07	24.2
......	.027	.14	19.3
17.5	.030	.14	21.4
13.0	.037	.15	24.6
15.0	.059	.24	24.5
10.0	.020	.10	20.0
11.0	.021	.09	23.3
12.0	.026	.13	20.0
15.0	.036	.15	24.0
12.0	.021	.11	19.1
15.6	.032	.14	23.5
Average..12.8	.029	.134	21.3

[1] Cc of N/10 acid per 100 grams (Methyl Orange, indicator).

Inspectors in the western district of the bureau sent to the Denver laboratory, during the summer of 1914, a number of salted and unsalted butters made from unneutralized cream. Samples of salt used in their manufacture also were analyzed. These samples were obtained from the States of Washington, Oregon, Colorado, New Mexico, and Arizona, and can be regarded as authentic. The data obtained are given in Table 8.

TABLE 8.—*Analyses of salted and unsalted butters made from unneutralized cream. 1914.*

Salt.	Salt-free ash.	CaO in butter.	CaO in salt-free ash.	Alkalinity.[1]	CaO in salt.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*		*Per cent.*
......	0.102	0.021	20.5	9.0	....
2.45	.126	.025	20.2	12.0	0.105
......	.092	.015	16.3	7.5	....
2.72	.134	.025	18.6	12.0	.370
......	.106	.021	20.1	9.5	....
4.28	.116	.020	17.2	11.0	0.0
......	.108	.026	23.9	10.5	....
3.49	.124	.031	25.0	13.5	.406
2.63	.114	.026	22.8	12.5	.360
......	.079	.018	22.7	8.0	....
4.85	.135	.030	22.2	12.0	.20
......	.075	.018	24.0	7.5	....
2.26	.137	.030	22.2	9.5	Unknown.
2.67	.140	.022	15.7	13.5	.193
1.78	.072	.018	25.0	9.5	.120
3.04	.088	.022	25.0	13.0	.140
......	.095	.021	22.1	9.5	....
3.87	.098	.022	23.0	10.0	Unknown.
......	.066	.015	22.7	7.0	....
......	.066	.015	22.7	7.0	....
0.56	.124	.025	20.1	13.0	.162
2.65	.118	.022	18.6	12.0	Unknown.
......	.076	.017	22.3	5.5	....
3.25	.100	.017	17.0	11.0	.056
......	.121	.027	22.3	11.0	....
2.90	.123	.025	20.3	14.0	0.0
......	.100	.019	19.0	10.0	....
2.53	.101	.018	17.8	11.0	.064
......	.106	.023	21.7	9.5	....
1.99	.123	.022	17.8	12.5	.078
3.71	.113	.022	19.4	11.5	.143
......	.098	.016	16.7	9.0	....
5.11	.136	.031	22.8	13.0	.147
......	.116	.026	22.8	10.0	....
Average. 2.98	.107	.022	20.9	10.8	.159

[1] Cc of N/10 acid per 100 grams (Methyl Orange, indicator).

It will be seen from the foregoing table that the calcium oxid in the salt-free ash of butter made from unlimed cream averaged about 21 per cent. This is believed to be a much more reliable determination than is either the alkalinity or the calcium oxid percentage in the entire sample. The alkalinity will vary through a rather wide range in butter, depending upon the care with which the buttermilk is removed. The amount of calcium oxid in the ash of the entire sample will vary with the quantity of buttermilk left in the butter and with the extent of calcium impurities in the salt. On the other hand, the percentage of calcium oxid in the salt-free ash is almost the same irrespective of how completely the buttermilk is removed. In order to test this point, two creams obtained from reliable sources were churned in the laboratory. One was well washed and the other not, the object being to obtain in one case a butter of low ash content and in the other case one of high ash content.

TABLE 9.—*Analyses of samples of two unneutralized creams and of the butter and buttermilk made from them.*

Determination.	Cream.		Butter.		Buttermilk.	
	Sample No. 1.	Sample No. 2.	Sample No. 1.	Sample No. 2.	Sample No. 1.	Sample No. 2.
Fat (per cent)	34.00	25.60				
Ash (per cent)			0.136	0.29	0.715	0.787
Ash on fat-free basis (per cent)	0.75	0.83				
Alkalinity of ash (cc N/10 acid per 100 grams)			15.60	26.40	68.00	73.20
Alkalinity on fat-free basis (cc N/10 acid per 100 grams)	76.50	78.40				
Calcium oxid in entire sample (per cent)			0.032	0.068	0.165	0.186
Calcium oxid on fat-free basis (per cent)	0.181	0.193				
Calcium oxid in ash (per cent)	24.10	23.20	23.50	23.40	23.00	23.80
Moisture (per cent)			15.44	29.80		
Curd (per cent)				1.60		

The results of analysis show that the two creams were normal. The composition of the buttermilk was very similar to that of the cream calculated to a fat-free basis. Calculating the analysis of butter No. 2 to the maximum percentage of moisture allowed (16 per cent),[1] gives:

Moisture (per cent)	16.00
Curd (per cent)	2.97
Ash (per cent)	0.54
CaO in entire sample (per cent)	0.126
CaO in ash (per cent)	23.40
Alkalinity of ash (cc N/10 acid per 100 grams)	49.10

The data presented in Table 9 show that while the percentage of calcium oxid and the alkalinity of the ash may vary considerably in different samples, depending upon conditions, and are therefore unreliable for detecting the practice of liming, even extreme conditions, as in butter No. 2, have no influence on the percentage of calcium oxid in the ash.

INFLUENCE OF LIMING ON COMPOSITION OF DAIRY PRODUCTS.

During the summer of 1913 and the winter of 1914 experiments were conducted in Denver in order to ascertain the influence of liming on the composition of cream, butter, and buttermilk.

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[1] U. S. Dept. of Agr., Office of the Secretary, Circ. 19, Standards of Purity for Food Products, p. 7. U. S. Internal Revenue Regulations No. 9, Revised July, 1907, p. 87, state that butter having 16 per cent or more of moisture is classed as adulterated butter.

EXPERIMENTS MADE DURING 1913.

The following experiment was conducted on a commercial scale at one of the Denver creameries in the summer of 1913:

Four hundred gallons of sour cream, weighing 3,230 pounds, were poured into the "dump box." The cream was well mixed by a revolving spiral and a sample of the raw, unpasteurized material was taken from the box. Approximately half of the cream was removed and pasteurized and a sample secured as it ran into the cooling tank. The remainder was treated with slaked lime mixed to a thin paste with water. The proportion of this paste added was about 1 quart to 100 gallons of cream. Samples representing the limed and unlimed parts of the same original lot were taken. The two creams were then run into separate tanks, properly cooled, and transferred into churns. The tanks were washed out with an unknown quantity of water from a hose and this water added to the cream in the churn. After churning, the butter was washed with water and properly drained. Samples were taken of the unsalted and salted butters and of the buttermilks made from the two varieties of cream. Samples of the salt used in the manufacture of the butters also were secured. This work was repeated in part at a later date. Two hundred gallons of cream were dumped, 1 quart of lime mixture added, and the cream pasteurized. Samples of the cream, before and after liming, and of the butter and buttermilk made from the limed cream were collected. The results of the analyses of these samples are given in Table 10.

TABLE 10.—*Analyses of unlimed and limed dairy products. Denver, summer of 1913.*

Description of sample.	Acidity (phenolphthalein, indicator).	Fat.	Ash.	Ash on fat-free basis.	Salt.	Salt-free ash.	Alkalinity of ash (Methyl Orange, indicator).	Alkalinity of ash on fat-free basis.	CaO in entire sample.	CaO on fat-free basis.	CaO in salt-free ash.	CaO in ash.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Cc N/10 acid per 100 grams.*	*Cc N/10 acid per 100 grams.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Unlimed cream:												
Before pasteurization	0.62	35.70	0.45	0.70				70.00	0.105	0.163		23.30
After pasteurization	.60	36.30	.47	.73				74.50	.112	.175		23.80
From dump box, experiment No. 2	.54	37.90	.455	.73				75.60	.115	.185		25.20
Limed cream:												
Before pasteurization	.43	35.70	.59	.91				110.40	.190	.295		32.20
After pasteurization	.32	35.00	.57	.87				103.10	.187	.287		32.80
Experiment No. 2	.40	37.30	.54	.86				96.40	.165	.263		30.30
Butter from unlimed cream:												
Unsalted		82.82	.108				8.00		.023			21.40
Salted		79.16	4.276		4.14	0.136	10.40		.030		22.00	
Butter from limed cream:												
Unsalted		83.00	.122				11.20		.034			27.80
Salted		77.74	4.544		4.39	.154	12.80		.042		27.20	
Experiment No. 2		84.36	.14				13.20		.038			27.10
Buttermilk from unlimed cream	.70		.71				60.50		.147			20.70
Buttermilk from limed cream	.48		.76				82.00		.225			29.60
Experiment No. 2	.55		.84				84.00		.225			26.60
Salt used in butter									.190			

This experiment shows that when lime is added to the raw material there is a considerable rise in the alkalinity of the ash and in the percentages of ash, calcium oxid, and calcium oxid in the ash, of both the limed cream and the resulting butter. In comparison with the values given in previous tables the percentage of calcium oxid in the ash or salt-free ash, as the case may be, seems most significant. Although the greater part of the added lime is removed in the buttermilk as calcium lactate, enough remains to cause a very appreciable increase in the percentage of calcium oxid in the salt-free ash. The analyses of the buttermilks do not correspond entirely to those of the respective creams on the fat-free basis because of the dilution with water, but the resemblance is very noticeable.

EXPERIMENTS MADE DURING 1914.

During the winter of 1914 additional data were obtained bearing on the composition of limed dairy products. These figures differ from those given in Table 10, for the reason that they were obtained from cream delivered in the winter. This cream, for obvious reasons, is not subject to spoilage in the same degree as commercial cream produced in the summer season and, consequently, its acidity is lower. It was desired to ascertain whether the addition of small amounts of lime could be detected. In the tabulated data the amount of lime added to the cream is not stated, because, in accordance with the usual practice, it was not gauged, but was stirred with water after slaking and then poured into the sour cream, the operator being guided by experience and by his sense of taste and smell as to the quantity necessary. The volume of liquid added, therefore, is no criterion of the quantity of lime introduced. That the quantity added was very small is shown by the slight reduction of the acidity. The data obtained are given in Tables 11 and 12.

TABLE 11.—*Analyses of unlimed and limed dairy products. Denver, winter of 1914. Plant No. 1.*

Product.	Acidity (phenolphthalein, indicator).	Fat.	Ash.	Ash on fat-free basis.	Salt.	Salt-free ash.	Alkalinity of ash (Methyl Orange, indicator).	Alkalinity of ash on fat-free basis.	CaO in entire sample.	CaO on fat-free basis.	CaO in salt-free ash.	CaO in ash.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Cc N/10 acid per 100 grams.*	*Cc N/10 acid per 100 grams.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Unlimed products:												
Cream, raw	0.34	35.80	0.447	0.696			44.00	68.50	0.105	0.164		23.30
Starter	.72	1.20	.765	.774			71.50	72.30	.183	.185		23.90
Cream and starter	.37	32.50	.474	.702			45.00	66.60	.112	.166		23.70
Unsalted butter			.120				11.00		.026			22.00
Salted butter			5.130		5.01	0.12	9.00		.027		22.50	
Buttermilk	.51	.60	.645				58.00		.152			23.60
Salt									.177			
Limed products:												
Cream, raw	.35											
Cream, limed, No. 1	.30	35.10	.456	.702			46.50	71.60	.116	.178		25.30
Starter No. 1	.72	1.20	.765	.774			71.50	72.30	.183	.185		23.90
Starter No. 2		1.15	.764	.772			73.00	73.80	.183	.185		23.90
Limed cream No. 1 plus starter No. 1	.34	31.10	.495	.719			47.50	68.90	.127	.184		25.60
Limed cream plus starter No. 2[1]		26.50	.580	.789			61.50	83.60	.162	.220		27.90
Unsalted butter No. 1			.140				13.50		.035			25.00
Unsalted butter No. 2			.145				14.00		.041			28.20
Salted butter No. 1			4.790		4.67	.12	11.50		.031		25.80	
Salted butter No. 2			4.940		4.83	.11	14.00		.032		29.00	
Buttermilk No. 1	.58	.60	.707				66.00		.177			25.00
Buttermilk No. 2		.75	.761				78.00		.215			28.20
Salt									.177			

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[1] Samples of raw and limed cream before the addition of starter were not obtained.

TABLE 12.—*Analyses of unlimed and limed dairy products. Denver, winter of 1914. Plant No. 2.*

Product.	Acidity (phenolphthalein, indicator).	Fat.	Ash.	Ash on fat-free basis.	Salt.	Salt-free ash.	Alkalinity of ash (Methyl Orange, indicator).	Alkalinity of ash on fat-free basis.	CaO in entire sample.	CaO on fat-free basis.	CaO in salt-free ash.	CaO in ash.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Cc N/10 acid per 100 grams.*	*Cc N/10 acid per 100 grams.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Unlimed products:												
Cream, pasteurized	0.31	35.60	0.434	0.683			39.00	60.50	0.101	0.157		23.20
Starter	.81	4.20	.750	.780			69.00	72.00	.189	.197		25.20
Cream plus starter	.36	33.20	.447	.669			42.50	63.60	.106	.158		23.70
Unsalted butter			.120				12.00		.024			20.00
Salted butter			5.370		5.25	0.12	13.50		.023		19.10	
Buttermilk	.45	.60	.669				57.00		.150			22.40
Salt									Trace.			
Limed products:												
Cream, raw	.38	37.60	.460	.73			38.00	60.90	.102	163		22.40
Cream, limed	.30	38.00	.465	.75			45.50	73.30	.129	.208		27.70
Starter	.89	4.00	.747	.77			74.00	77.10	.181	.188		24.30
Limed cream plus starter	.39	34.00	.509	.77			50.00	75.70	.136	.206		26.70
Unsalted butter			.150				13.00		.041			27.30
Salted butter			5.040		4.96	.08	9.50		.022		28.10	
Buttermilk [1]	.48	.20	.890				71.50	72.60	.198			22.20
Salt									Trace.			

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[1] This buttermilk has an undue amount of ash and a low percentage of calcium oxid in ash. The alkalinity of the ash and the percentage of CaO in the entire sample are normal. The sample may have been contaminated with a little ash material such as salt.

The maximum limits previously given for alkalinity and calcium oxid in unneutralized cream were exceeded in these two plants in only one and two cases, respectively. These limits, therefore, can not be depended upon to detect slight liming. The percentage of calcium oxid in the ash, however, shows clearly the addition of lime to the cream, even when the reduction of acidity of the cream, due to lime, is only 0.05 per cent.

As the same salt was used in both the limed and unlimed butter at each plant, any effect that the impurities of the salt might have would apply in the same degree to both kinds of butter.

ANALYSES OF BUTTER PROBABLY MADE FROM NEUTRALIZED CREAM.

In Table 13 are given a number of analyses of butters which, interpreted in the light of experimental data so far obtained, are believed to have been made from limed cream. Some of the analyses represent butter made by creameries which admit that they use lime as a neutralizer. Those marked with an asterisk are authentic samples collected by inspectors of the bureau.

TABLE 13.—*Analyses of butters believed to have been made from limed cream.*

Salt.	Salt-free ash.	CaO in butter.	CaO in salt-free ash.	Alkalinity (Methyl Orange, indicator).	CaO in salt.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Cc N/10 acid per 100 grams.*	*Per cent.*
*3.45	0.131	0.045	34.3	14.0	0.142
*3.99	.101	.026	26.2	11.5	.085
*3.25	.238	.096	40.3	28.0	.192
*......	.206	.090	43.8	25.0	....
......	.100	.032	32.0	14.0	....
......	.140	.041	29.6	14.7	....
......	.150	.045	30.0	17.5	....
......	.100	.030	30.0	17.5	....
......	.110	.035	31.8	17.5	....
......	.140	.040	28.5	15.0	....
......	.120	.033	27.5	12.0	....
......	.140	.054	38.5	20.0	....
......	.180	.056	31.1	17.0	....
Average....	.142	.048	32.5	17.2	....

It can be seen readily from the above figures that the butters represented by these analyses are of a very different character from those given in Tables 7 and 8 and from the unlimed samples recorded in Tables 10, 11, and 12. An inspection of the various tables will show that although the determinations of alkalinity and of calcium oxid in the entire sample may indicate liming, they can not be relied on altogether, as the figures for limed and unlimed butter will often overlap, depending on factors met with in manufacturing. The percentage of calcium oxid in the ash or in the salt-free ash will reveal the liming, even if it be but slight.

EFFECT OF IMPURITIES IN THE SALT ON THE PERCENTAGE OF CALCIUM OXID IN THE SALT-FREE ASH.

CALCIUM SULPHATE.

The most usual impurity in dairy salt and the one present in greatest quantity is calcium sulphate. The amount of salt-free ash in butter will increase with the calcium content if salt containing calcium sulphate is used, but it will not increase in the same ratio. The calcium oxid percentage in calcium sulphate is 41, in the ashes of dairy products it averages about 22 per cent. This difference will cause an increase in the calcium oxid percentage in the salt-free ash, the amount of increase depending upon the quantity of salt used and the amount of impurity in it.

Street[1] found in 21 samples of salt examined, from 0.30 to 1.23 per cent of calcium sulphate and from 0 to 0.35 per cent of calcium chlorid. Woll[2] found in domestic brands of dairy salt percentages of calcium sulphate ranging from 0.31 to 1.87 per cent and of calcium chlorid from 0.02 to 0.65. The maximum allowances for table or dairy salt that have been established by the Department of Agriculture are: 1.4 per cent for calcium sulphate and 0.5 per cent for calcium chlorid plus magnesium chlorid.[3]

To ascertain the effect of impurities in the salt upon the percentage of calcium oxid in the salt-free ash, a number of experiments were made. Four unsalted stock butters, A, B, C, and D, were used for this purpose. About 5 pounds of butter were melted and well shaken during solidification. Portions of 200 grams were withdrawn and melted in a bottle with 10 grams of salt of known composition. The butter and salt were well mixed by shaking during solidification. The butter treated in this manner contained approximately 4.75 per cent of salt. This is more salt than the average American creamery butter contains, but it was thought best to work with quantities higher than the average so that a certain amount of margin for variation might be provided.

The methods employed in the analysis of butter thus treated were the same as those given on page 3. Figures for sulphur trioxid, calculated to calcium sulphate, are also included. In some cases the sulphate was determined by dissolving the ash of 40 grams of butter in dilute hydrochloric acid and then precipitating with barium chlorid. In other cases 100 grams of butter were melted and extracted with 50 cc portions of warm water acidulated with hydrochloric acid. The water extract was cooled and made up to 250 cc, after adding some pure cupric chlorid or mercuric bichlorid solution to precipitate the proteins. Two hundred cc of the clear filtrate were treated with barium chlorid as in the usual sulphate determination. The results are given in Table 14.

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[1] Street, J. P. Thirteenth Report on Food Products. *In* Conn. Agr. Exp. Sta. Biennial Rpt., 1907–08, Pt. IX, p. 595.

[2] Woll, F. W. A Study of Dairy Salt. *In* Wis. Agr. Exp. Sta. Bul. 74, pp. 12–13, May, 1899.

[3] U. S. Dept. Agr., Office of the Secretary, Circ. 19, Standards of Purity for Food Products, p. 19.

TABLE 14.—*Analyses of butters mixed with salt of known composition, showing the effect of impurities on the percentage of calcium oxid in the salt-free ash.*

Stock sample.	Salt added to butter.	Composition of salt.	CaO in butter.	SO_3, calculated as $CaSO_4$[1].	SO_3, calculated as $CaSO_4$[2].	Salt-free ash.	CaO in salt-free ash.	CaO in salt-free ash corrected for $CaSO_4$ in salt[3].	CaO in salt-free ash corrected for $CaSO_4$ in salt[4].
	Grams to 100 grams.	*Per cent.*	*Per ct.*	*Per cent.*	*Per cent.*	*Per ct.*	*Per ct.*	*Per cent.*	*Per cent.*
A	0	....	0.0177	0.002	....	0.087	20.3	....	....
B	0	....	.0275	....	....	.139	19.7	....	....
C	0	....	.0400	.005	....	.204	19.6	....	....
[5]D	0	....	.0504	....	....	.150	33.6	....	....
A	5	C. P.	.0170	.005	....	.099	17.1	....	....
B	5	do	.0250	....	....	.134	18.6	....	....
C	5	do	.0366	.003	....	.176	20.8	....	....
D	5	do	.0460	....	....	.142	32.4	....	....
B	1	do	.0252	....	....	.142	17.7	....	....
C	1	do	.0372	....	....	.179	20.8	....	....
A	5	0.296 $CaSO_4$	.0238	.014	....	.121	19.7	17.0	....
A	5	0.505 $CaSO_4$	.0282	....	....	.129	21.6	17.4	....
C	5	0.505 $CaSO_4$	.0500	.014	....	.197	25.3	24.1	....
A	5	1.067 $CaSO_4$	.0383	....	....	.162	23.6	....	....
C	5	1.067 $CaSO_4$	.0587	.037	....	.218	26.7	23.7	....
A	5	1.427 $CaSO_4$	.0452	.063	....	.173	25.5	17.3	....
B	5	1.427 $CaSO_4$	.0550	.061	0.070	.202	27.2	21.2	19.7
C	5	1.427 $CaSO_4$	.0640	.055	....	.231	27.7	23.4	....
A	5	2.097 $CaSO_4$	.0565	.094	....	.201	28.1	16.7	....
B	5	2.097 $CaSO_4$	.0665	.090	.103	.229	29.0	21.1	19.2
C	5	2.097 $CaSO_4$	.0768	.079	....	.262	29.3	24.2	....
B	2.5	2.097 $CaSO_4$	.0443	.034	....	.184	24.0	20.1	....
C	5	0.20 $CaCl_2$	.0421	....	....	.177	23.7	....	....
B	5	0.42 $CaCl_2$	.0352	....	....	.140	25.1	....	....
C	5	0.42 $CaCl_2$	.0480	....	....	.181	26.5	....	....
B	5	0.63 $CaCl_2$	.0410	....	....	.144	28.5	....	....
C	5	0.63 $CaCl_2$	.0524	....	....	.185	28.3	....	....
B	2.5	0.63 $CaCl_2$	.0333	....	....	.139	24.0	....	....
B	5	1.43 $CaSO_4$ 0.47 $CaCl_2$	.0620	.062	.072	.207	30.0	25.1	24.0
B	2.5	1.43 $CaSO_4$ 0.47 $CaCl_2$	.0440	.025	....	.174	25.1	22.6	....
D	5	1.00 Na_2SO_4	.0435	....	....	.172	25.3	....	....
D	5	0.93 $MgCl_2$	.0443	....	....	.148	30.0	....	....
[6]D	5	[6]1.22 $CaCl_2$ 0.51 $MgCl_2$	.0643	....	....	.141	45.6	....	....

[1] Determined in ash.

[2] Determined in water extract.

[3] Determined from SO_3 in ash.

[4] Determined from SO_3 in water extract.

[5] Sample D was a butter made from a limed cream.

[6] A visible precipitate of $MgNH_4PO_4$ was obtained from the ash of 20 grams of butter from 100 cc of solution.

An inspection of Table 14 reveals a number of interesting points. The first one noted is the change in the percentage of calcium oxid in salt-free ash when chemically pure salt is added. This change seems to depend upon the quantity of butter ash. If the quantity present is low the change is minus, becoming a plus with the higher quantities of ash. The reason for this has not been determined but it is probably due to conditions of ashing. With higher quantities of ash this action seems to decrease materially. Since this change would, in the majority of cases, give the benefit of any doubt to the manufacturer, it is not thought to be of any serious consequence.

Table 14 also shows that relatively high percentages of salt, contaminated to a considerable degree, must be added before the percentage of calcium oxid in the salt-free ash of butter made from unneutralized cream will exceed 25, the tentative maximum limit. If only 2.5 per cent of salt, the average quantity present in American butter, is added, the salt may contain about 2 per cent of calcium sulphate before the limit is reached. Furthermore, a correction for calcium sulphate in the salt can be applied by determining the sulphate in the butter. The sulphate found is calculated to calcium sulphate and this figure subtracted from the salt-free ash. The equivalent amount of calcium oxid is subtracted from the total calcium oxid, and the quotient of these corrected figures gives the actual percentage of calcium oxid in the salt-free ash. The determination of the sulphate in the acidulated water extract is preferable to the ashing method, as the results are higher owing to the well-known action of phosphorus pentoxid in driving out sulphur trioxid unless sufficient alkali is present. This action is especially strong in sample C, because of its higher ash and consequently higher phosphorus pentoxid content. It is possible that better results would be obtained by the ashing process if sodium carbonate were first added, but since the extracting process is just as quick, it appears to be preferable. The amount of sulphates in the ash of unsalted butter or butter salted with chemically pure salt, derived from the sulphur of the casein, is so slight as to be negligible. In the extract method it would be removed by the protein precipitant.

CALCIUM CHLORID.

When salt is contaminated with calcium chlorid the action is somewhat different. The calcium chlorid does not increase the salt-free ash because it is almost equivalent to the two sodium chlorid molecules calculated from the silver chlorid precipitate. Therefore, the calcium oxid is increased without corresponding increase in the salt-free ash, causing an increase in the percentage of calcium oxid in the salt-free ash. With about 5 per cent of salt in the butter it requires an impurity in the salt of approximately 0.4 per cent calcium chlorid before the percentage of calcium oxid in the salt-free ash of the butter will reach the limit of 25. Salt to the amount of 2.5 per cent, when added to butter, may contain about 0.65 per cent of calcium chlorid, the maximum amount in dairy salt as found by Woll,[1] without causing errors in interpretation. No correction for calcium chlorid can be applied.

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[1] Woll, F. W. A Study of Dairy Salt. Wis. Agr. Exp. Sta. Bul. 74, p. 13, May, 1899.

Analyses made by the water laboratory of the Bureau of Chemistry of samples of salt collected from representative markets of the United States showed but very small percentages of calcium chlorid, magnesium chlorid, or sodium sulphate, the main impurity being calcium sulphate. However, within a radius of 200 miles from Cincinnati, a salt is produced and consumed largely locally that contains no sulphate and relatively large percentages of calcium and magnesium chlorids ($CaCl_2$, 0.9 to 2.3 per cent; $MgCl_2$, 0.3 to 0.8 per cent). Table 14 shows that the effect of adding this salt to butter is to increase greatly the percentage of calcium oxid in the salt-free ash. An unneutralized butter might, therefore, occasionally be classed as neutralized if highly salted with salt contaminated with considerable calcium chlorid. Analyses of butter, particularly if the butter is produced in the above locality, must be scrutinized with special care if they show no sulphate. Magnesium chlorid is associated with calcium chlorid in Ohio salt, and an increase in the magnesium oxid percentage in butter salted with it might be expected. Leach[1] states that the magnesium oxid content of the ash of milk is only 2.42 per cent. The maximum amount of magnesium oxid that could be present in 50 grams of butter, therefore, would be only about 2 milligrams. Any increase could be regarded as due to impurities in the salt, and a high percentage of calcium oxid in the salt-free ash would thus be explained. However, this salt is to be found only in a very limited area, and the amount of butter affected thereby is exceedingly small compared with the total product of the United States. The effect of calcium chlorid, therefore, may be considered practically negligible except in a very few special cases.

CALCIUM SULPHATE AND CALCIUM CHLORID.

If both calcium sulphate and calcium chlorid are present, their effect is cumulative. In Table 14 is shown a case where about 5 per cent of salt which contained the maximum amount of calcium sulphate and calcium chlorid allowed in dairy salts[2] was used in salting the butter. The percentage of calcium oxid in the salt-free ash reached 30, but when the effect of the calcium sulphate was calculated, though retaining the effect of the calcium chlorid, it dropped to 24, 1 per cent below the maximum. When 2.5 per cent of this salt of maximum allowed impurity was employed, the percentage of calcium oxid in the salt-free ash was only 25, without any correction for calcium sulphate.

MAGNESIUM CHLORID.

Should salt containing magnesium chlorid be used the percentage of calcium oxid in the salt-free ash of the butter would be lowered because of an increase in the salt-free ash without a corresponding increase in the calcium oxid in the butter. In the ashing process the magnesium chlorid is decomposed with loss of chlorin and the salt-free ash is increased by the equivalent magnesium oxid. This point is well illustrated in Table 14. But it would be only in the case of a butter salted with large quantities of salt of exceptionally high magnesium chlorid content that a serious lowering of the calcium oxid determination would result in a butter made from neutralized cream being classed as made from unneutralized cream.

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[1] Leach, Albert E., Food Inspection and Analysis, 3d ed., p. 128, New York, 1913.

[2] U. S. Dept. of Agriculture, Office of the Secretary, Circ. 19, Standards of Purity for Food Products, p. 19.

SODIUM SULPHATE.

The use of salt contaminated with compounds other than calcium chlorid or calcium sulphate causes a lowering of the percentage of calcium oxid in the salt-free ash. Sodium sulphate, for example, increases the salt-free ash without increasing the calcium oxid. If it were known that the salt used contained sodium sulphate its effect could be calculated as easily as that of calcium sulphate. Fortunately, the amount of impurities other than calcium salts in American dairy salt is small and their effect is almost negligible.

INTERPRETATION OF RESULTS.

The figure 25 as the maximum percentage of calcium oxid in the salt-free ash of unneutralized butter is considered a good working standard. It allows considerable latitude for the effect of calcium impurities in the salt. If the CaO figure of any particular butter is below 25, the butter may immediately be classed as made from unlimed cream.

If the brand of salt used in the manufacture of any given sample of butter could be ascertained the interpretation of results of analysis would be greatly simplified. Analyses of the most widely used brands of American dairy salt would be useful in connection with this work.

Butters with a percentage of calcium oxid in the salt-free ash of between 25 and 28 should be classed as suspicious. After correction has been made for calcium sulphate, determined from the sulphate present, if the percentage of calcium oxid in the salt-free ash is still above 25, the butter was made from a limed cream. If the CaO figure drops from above 25 to below 25, the apparently high percentage was caused by the calcium sulphate in the salt used, and the butter must be considered as made from unlimed cream. A butter which contains more than 28 per cent of calcium oxid in the salt-free ash, especially when the percentage of salt or sulphate is low, can be identified as made from a neutralized cream. A sulphate determination should be made on all butters which show a CaO figure above

25, and the proper correction applied. It may be assumed safely that all the sulphate is present as calcium sulphate, as the amount of sulphates other than calcium in salt is usually very small. Correction may not always give absolutely true values, but in the majority of cases the results are probably well within the limits of error of the work. The limit of 25 is not to be considered as an arbitrary rule that may not be altered by future work, but rather as a fair maximum standard, especially after correction is made for calcium sulphate in the salt, and one that should give the manufacturer the benefit of any doubt.

USE OF LIME TO RENOVATE OLD STORAGE BUTTER.

While cooperating with the Denver internal revenue office another use of lime as a neutralizer was detected. Upon applying the spoon test for renovation on some samples of butter submitted by the revenue agent, a very peculiar reaction was noticed. The butter when heated formed a foam similar in appearance to that observed when glucose is heated for ashing purposes. Instead of forming large bubbles which broke quickly, these samples produced a foam with very small bubbles, which held up for an unusual length of time and emitted a strange, sweetish odor when heated. Upon factory inspection it was found that the firm making the product examined was using lime to sweeten and deodorize old storage butter. It was essentially a chemical renovation process. Frozen storage butter was broken into a tank containing slaked lime and water and was allowed to "temper" for about 12 hours. The product was then removed, washed, worked over, and sold as creamery butter.

In Table 15 are given the data collected on this variety of butter.

TABLE 15.—*Analyses of storage butters renovated by the use of lime.*

Sample. No.	Alkalinity.	CaO in butter.	Salt-free ash.	CaO in salt-free ash.
	Cc N/10 acid per 100 grams.	*Per cent.*	*Per cent.*	*Per cent.*
3053-E	102.0	0.282	0.40	70.5
3054-E	26.6	.069	.15	46.0
3055-E	62.0	.174	.29	60.0
3057-E	21.5	.069	.19	36.3
3063-E	29.0	.085	.20	42.5
3064-E	....	.080	.20	40.0
5533-E	67.0	.180	.32	56.2
5534-E	68.0	.175	.31	56.4
5553-E	97.5	.275	....	....
5554-E [1]	297.0	.840	....	....
5555-E [1]	341.0	.940	....	....
5566-E	26.5	.078	.17	45.8
5567-E	29.0	.083	.18	46.1
5568-E	....	.190	.33	57.5
5569-E	....	.146	.25	58.4
5570-E	28.5	.080	.17	47.0
5571-E	28.5	.086	.21	40.9

[1] Butter mixed with lime water taken from tank in which the butter was neutralized.

All the butters, the analyses of which are given in Table 15, were alkaline to litmus to a greater or less degree, and had a peculiar foam test and a sweetish smell on heating, especially those of high lime content. When heated with dilute sulphuric acid all of them gave a more or less distinct smell of butyric acid, thus indicating the presence of calcium butyrate. The water extracts of these butters were alkaline to litmus paper and those containing the larger quantities of calcium oxid were alkaline to phenolphthalein. Some of these samples have been kept for months in an ice-box without developing the characteristic smell of rancid butter, although many became moldy and possessed a bad odor. When the butter was treated with hot alcohol, as in the determination of acids in fats, considerable acidity was usually found, indicating the presence of free oleic and palmitic acids.

The liming of cream is a renovation of a deteriorated intermediate product, while the liming of butter is a renovation of a more or less spoiled final product. The chemistry of the two processes is different. The main acid constituent of sour cream is lactic acid, and when lime is added calcium lactate is probably the principal substance formed, but with an excess of lactic acid remaining. When this cream is churned the percentage of calcium oxid in the salt-free ash is increased because of the quantity of calcium lactate remaining in the butter fat. It is probable that with excessive washing these calcium salts could be washed out, but with the calcium would go the flavor, a property the creamery man desires to keep in his product as its value depends upon it, in large measure. Since the lime is never added in excess, the butter will give a more or less acid reaction to litmus paper.

Storage butter is always acid even when not rancid. When it is placed in a lime solution the free water-soluble acids that may be present are neutralized to a greater or less degree. Since these acids are perhaps one of the main causes of bad odor in rancid butter, their neutralization will sweeten and deodorize the product. The nonvolatile, nonsoluble acids, oleic and palmitic, play only a secondary part in this process. These acids and their calcium salts are practically insoluble in water and would be neutralized only in small part. By using alcohol their presence in the free state can be demonstrated. The calcium salts of the offensive water-soluble volatile acids are more or less soluble in water and are removed partly in the subsequent washing, the amount which remains depending on the extent of the washing. Their presence can be demonstrated by their odor on adding dilute sulphuric acid. Nor is the excess of calcium hydroxid entirely removed, for all of the water extracts of these butters gave an alkaline reaction to litmus. When such butter is ashed the added calcium salts will cause an increase in the alkalinity of the ash, and especially in the percentage of calcium oxid in the salt-free ash. It should be noted, however, that the alkalinity of a butter made from limed cream, or even of one washed in lime water, might be less than that of a natural butter with a high whey content, but an increase in the percentage of calcium oxid in the salt-free ash would at once reveal the use of lime at some stage. Perhaps by determining the ratio of alkalinity to another constituent of the butter ash, for example, phosphorus pentoxid, an independent method might be developed for detecting neutralization. It might be necessary to distinguish between a butter made from limed cream and a butter given a "lime bath." If the alkalinity were below 20, the reaction of a water extract acid to litmus, and the percentage of calcium oxid in the salt-free ash, corrected for calcium sulphate in the salt, well above 25, the use of a limed cream would be indicated. If, on the other hand, a butter had an alkalinity of over 20, if the percentage of calcium oxid in the salt-free ash was over 35, if its water extract was alkaline to litmus, and if it gave a peculiar foam test and liberated butyric acid on the addition of dilute sulphuric acid, it would be strong evidence that lime had been added to the butter and not to the cream.

SUMMARY.

The percentage of calcium oxid in the ash of milk, unneutralized cream, and butter made from unneutralized cream varies within fairly narrow limits, with a tentative maximum set at 25.

When lime has been added to the cream in the process of manufacture this percentage is increased above 25, the increase varying with the amount of lime added and the degree of washing.

The effect of calcium impurities in the salt has been studied and a method developed to correct for the chief impurity, calcium sulphate.

It has been shown that the percentage of calcium oxid in the salt-free ash of butter made from unneutralized cream will not exceed the tentative maximum limit of 25, unless high percentages of salt with much impurity are employed.

Finally, attention has been called to a form of sophistication of butter by chemical renovation.

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